Experiment 4:
Distillation

Distillation is a method of identifying and purifying of organic compounds. For instance, the boiling point of a compound, and hence its identity, can be determined by distillation. Since a chemical's boiling point is a well-defined physical property, distillation can also be utilized to separate the solution as a means of purification, given that each of the components have different boiling points.

As a liquid solution is heated in a distillation apparatus, the vapour pressure of the liquid (the tendency for the molecules to escape from the surface) increases until it is equal to the atmospheric pressure. At this point the liquid is said to be boiling. Additional heat supplied by further boiling provides the energy necessary for conversion of the liquid phase to the gaseous phase. Distillation is a process of boiling a liquid to vapour, then re-condensing the vapour back to a liquid, called the distillate. The vapour will rise in the apparatus and, in time, warm the distillation head and the thermometer before flowing down the condenser. The cool walls of the condenser remove heat from the vapour and condense it back to a liquid form called the condensate, or distillate.

How are the walls of the condenser kept cool?

Why slow and steady?

"Distillate" and "condensate" may be used interchangeably.

*Distillations should always be carried out slowly and steadily to ensure that the liquid and vapour remain in equilibrium. When this situation has been attained, the column of vapour will rise slowly up to the thermometer bulb and a small drop of liquid will condense on the bulb. This drop of condensate should remain on the thermometer throughout the distillation, constantly bathed by the flow of vapour. The temperature of the vapour at the distillation head will then be the "true" boiling point of the liquid being distilled. If too much heat is applied, the liquid will be **superheated** and the equilibrium will be lost. As a result, the vapour composition will change, becoming richer in the component of the mixture with the higher boiling point. If superheating has occurred, the drop of condensate will disappear. Rapid distillation will carry the system away from equilibrium resulting in poor separation of the volatile components Therefore, keep in mind that <u>slower the distillation will yield better results.</u>*

This experiment is comprised of two parts. In the first part you will be performing simple distillation of an unknown liquid. From the boiling point obtained in your simple distillation, you can determine the identity of the unknown. In the second part of the experiment, you will carry out a fractional distillation on a solution of unknown and water to determine the percent composition of the unknown solution. If the solution does not form an azeotrope, then the liquid will distill at approximately the same temperature as the boiling point of the pure unknown. If an azeotrope forms, then the boiling point of the first fraction may, or may not be different. If the boiling point differs, this will be the first clue that you are distilling an azeotrope. You will also obtain your partner's results and analyze their data. Construct a graph of temperature versus distillate volume for each distillation (using your data, as well as your partner's) using the graph papers provided. Before coming to the lab, think about what this graph should look like. Have all four graphs initialled by your TA before leaving the lab. From the graphs, determine the boiling point of the unknown liquid, the identity of the unknown liquid, and the percent composition of your assigned solution. Enter the information on the Data Sheet provided and hand these all in with all the graphs at the beginning of your next lab period (<u>Before</u> the quiz!).

In this lab, you will be distilling one of the compounds listed below. You must check section D of the CRC Handbook (older editions in the lab are more useful for this) to find out which of these compounds may form an azeotrope, and the composition of any azeotrope that forms. You must do this before coming to the lab! Before beginning this experiment, read the section entitled "Liquids," found in the Appendix. Three basic types of distillation are outlined there. (We will not be doing a Co-distillation in this course.)

Name of Unknown	**Literature Boiling Point °C**
Methanol	64.7
1-Propanol	97
2-Propanol	82.4
Ethanol	78.3

Procedure – Simple Distillation

1. Since ground-glass joints are rigid, the apparatus must be assembled carefully to avoid any excess strain that can cause very costly fractures. First, obtain the necessary clamps (two with variable length capabilities and two ring clamps) and a heating mantle.
2. Next, take 25mL of the assigned **pure** unknown (A, B, C or D) and place it in a 50mL round bottomed flask. Then add some boiling chips.
3. Ensure that the outside of the flask is dry before placing it on the heating mantle. Remember to clamp around the neck of the RB flask, so the mantle can be lowered quickly in case of an emergency. Make sure that the flask has good contact with the mantle for maximum heating efficiency, and that the clamp length is adjusted so that the remaining apparatus will be vertical.
4. Lightly grease all ground-glass joints (your TA will show you how to do this).
5. Attach the distillation head to the RB flask, then twist the thermometer in place.
6. Place an elastic band around the bottom water inlet of your condenser, and attach the thin walled tubing to the glass.
7. Position an adjustable clamp to the bench stand in an approximately "correct" position to receive the condenser, while keeping the rest of the apparatus vertical.
8. Attach the condenser joint to the distillation head, while the clamp is slightly loose. If one hand is holding the condenser, the other can be moving and tightening the clamp. The joints should fit together well without force and there should be no possible leak. If the joints do not fit together properly, or the apparatus is no longer vertical, then check the height of the condenser clamp. Once this is done, tighten the clamp.
9. When you are satisfied, place the glass adapter on the end of the condenser, stretching the elastic from the bottom water inlet of the condenser to prevent the adapter from falling off the end of the apparatus. Make sure that all connections are tight, but do not force them.

The RBF should be filled no more than 2/3, and no less than 1/3 its capacity. Why?

The outside of the RBF should be dry before placing on the heating mantle. Why?

The glass joints of the apparatus should be greased. Why?

The RBF should be in good contact with the heating mantle. Pourquoi?

In the apparatus, what's the most important piece to be clamped, and why?

10. Place a graduated cylinder under the adapter to receive the distillate.

11. Ask your TA to check your apparatus before you proceed. Once your TA has approved your set up, turn on the faucet slowly (only a low flow is needed) then proceed heating on a high setting until boiling begins.

12. Once boiling has begun, adjust the heat on the heating mantle until the distillate **collects at a rate of about 1mL per 1.5-2 minutes (keep the temperature steady)**. Remember to regularly check that the drop of distillate has formed on your thermometer bulb.

13. In your notebook, record the temperature given on the thermometer and total volume collected at regular intervals (every 1mL of distillate formed).

14. When finished, turn off and lower the heating mantle to allow the flask to cool until it can be handled safety and discard any remaining liquid in the "chemical waste" bottle in your fume hood.

15. You can plot your graph while your fractional distillation is heating up.

During distillation, what's an appropriate height for the condensate in the condenser to reach?

How should the graph look in the end?

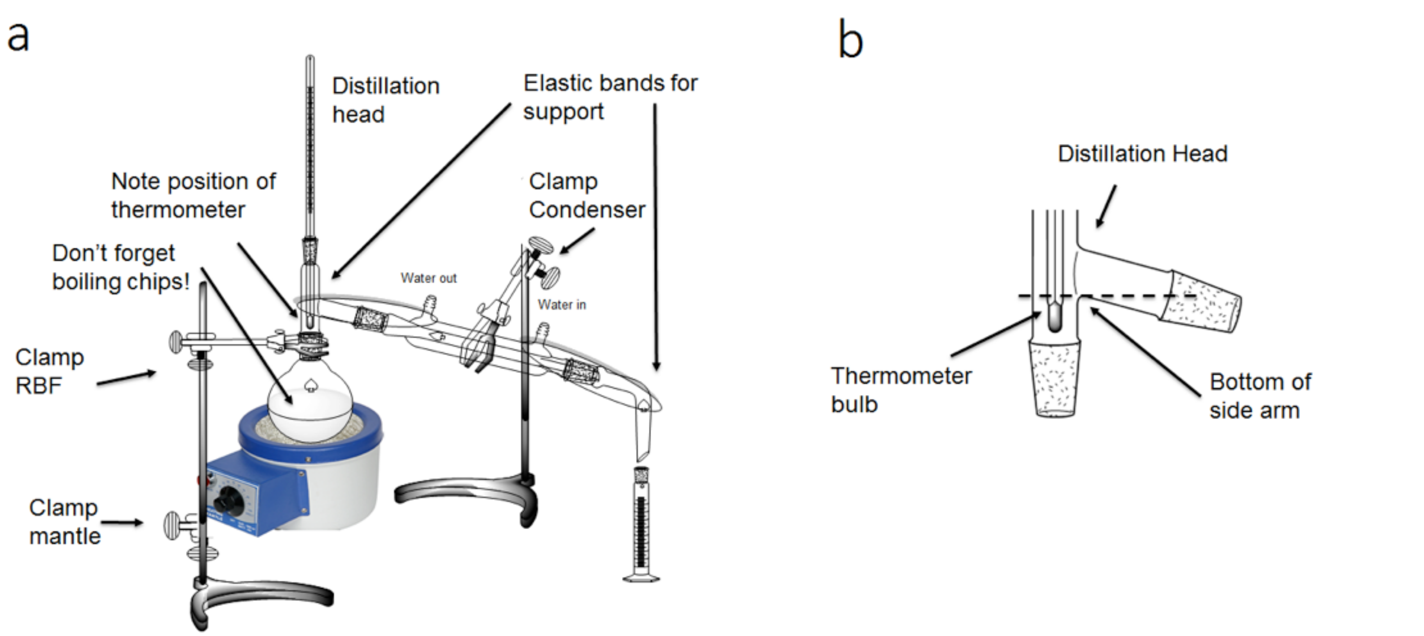


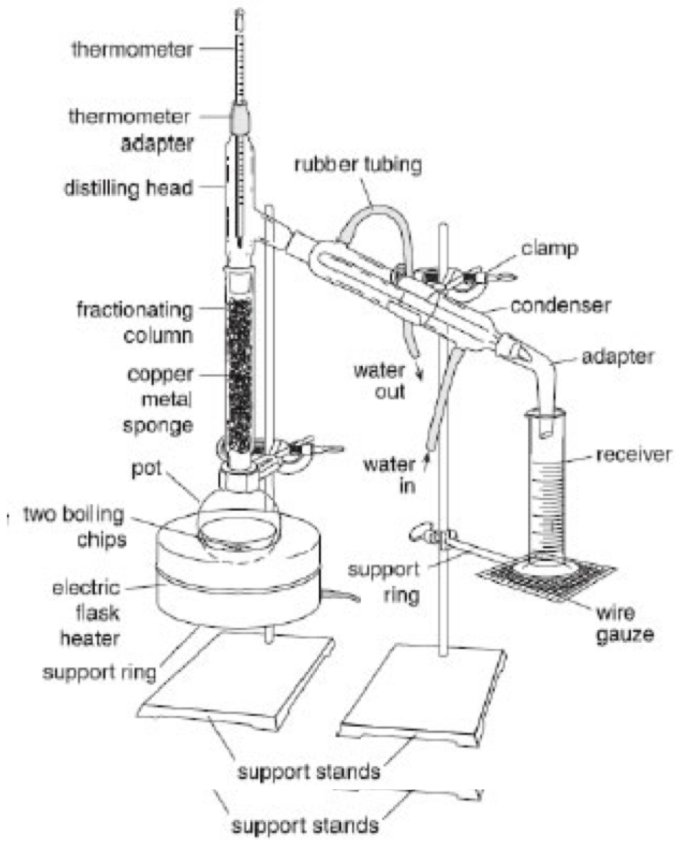
Figure 1. Simple distillation. a, Diagram of the simple distillation apparatus. b, Correct position of the thermometer inside the distillation head. The thermometer bulb must be entirely below the bottom of the side arm.

Procedure - Fractional Distillation

What's the purpose of the fractionating column and insulation?

1. Obtain 25 mL of your assigned unknown **solution** A, B, C, D, E or F (this is NOT the same sample that you used for simple distillation! It will be in a different bottle), and add boiling chips.
2. Set up the apparatus for fractional distillation (Refer to Figure 2 below)

Figure 2. Fractional Distillation Apparatus



3. **Insulate** the top of the RB flask and the fractionating column, *only up to the thermometer bulb*, with aluminum foil.
4. Again, ensure that all joints fit and that the fractionating column is vertical.
5. Have your demonstrator check the apparatus.
6. Begin heating and bring the solution quickly to a boil. As soon as boiling begins, turn down the heat supply, but maintain boiling. You will be able to feel the fractionating column heat

up slowly as a ring of condensate travels slowly upwards. Do not apply more heat until you are sure that the ring of condensate has stopped rising; the rise must be gradual so that the column can acquire a steady temperature gradient. If carried out properly, it will take several minutes for the condensate to reach the top of the column.

7. Once distillation has begun, it should continue steadily, without any rise or drop in temperature and at a rate **no greater than 1 mL in 1.5-2 minutes.** If no azeotrope forms, the temperature should be at the boiling point of the pure unknown; if an azeotrope does form, then the boiling point *may* differ.

8. Observe the flow and keep it steady by adjusting the heat setting to obtain equilibrium and the required results of a steady temperature.

9. From the boiling point obtained from your simple distillation, you should have figured out the identity of your unknown liquid. Keep this in mind when adjusting the mantle setting.

10. Record in your lab notebook the temperature as each 1 mL of distillate collects and graph your results. The temperature axis of the graph should begin a little below the first boiling point of the solution and end at the boiling point of water. What do you expect the graph to look like?

11. Make more frequent readings when the temperature begins to rise abruptly. Stop the distillation (turn off, & lower mantle) when a second constant temperature is reached. (What should that temperature be?).

12. When finished, discard the distillate and residues into the waste chemical bottle provided in the fume hood. Return used aluminum foil to the front cart.

What’s a theoretical plate? Why is it used? (Hint: Refer to the “Fractional Distillation” section in the Appendix!)

Questions on Distillation

1. Why should a distillation be done relatively slowly, rather than as quickly as possible?

2. Why is the position of the thermometer important in a distillation apparatus?

3. What would happen if the water ran through the condenser from the top to the bottom?

4. Why should a distilling flask at the beginning of a distillation be:

a) filled to not more than 2/3 of its capacity?

b) filled to not less than 1/3 of its capacity?

5. What is a theoretical plate?

For more details on this experiment, see the appendix of this lab manual.

DISTILLATION DATA SHEET **Name:** ____

TA: _________________________________ **PRA**______ **Rm**_______ **Space** ______

To be handed in with two sets of graphs. (one your own; one partner's)

1. **Your own Simple Distillation, Unknown** ______ , Boiling point ________ °C

   Show on your graph why you state the boiling point given above.

   Explain any difficulties with your results:

   From the boiling point, the Unknown liquid is ___________________________

   Explain (if necessary):

2. **Your own Fractional Distillation**

Mark, clearly with a "P", the position on the graph where your liquid was all distilled off.

State this amount here: _________ mL.

Assuming that the Unknown did not form an azeotrope, the beginning percent composition of Unknown ____/H_2O solution would be: (show calculations!)

3. From your conclusions in the Simple Distillation, you have identified your unknown. Check the CRC handbook, Section D, to find out whether this liquid does form <u>any</u> azeotropes. If so, list any azeotropes below:

If your Unknown did form an azeotrope with water, the beginning percent composition of Unknown ____/H_2O solution would be: (show calculations!)

DISTILLATION DATA SHEET **Partner's Name:** ____

TA: __________________________________ **PRA**______ **Rm**_______ **Space** ______

To be handed in with two graphs. [One your own, one of partner's]

1. **Partner's Simple Distillation, Unknown** _______ , Boiling point _______ ^{o}C.

   Show on your graph why you state the boiling point given above.
   Explain any difficulties with your results:

   From the boiling point, the Unknown liquid is ____________________.
   Explain (if necessary):

2. **Partner's Fractional Distillation**

Mark, clearly with a "P", the position on the graph where your liquid was all distilled off.
   State this amount here: _________ mL.

   Assuming that the Unknown did not form an azeotrope, the beginning percent composition of Unknown ____/H_2O solution would be: (show calculations!)

3. From your conclusions in the Simple Distillation, you have named identified your unknown. Check the CRC handbook to find out whether this liquid does form <u>any</u> azeotropes. If so, list any azeotropes below:

   If the Unknown did form an azeotrope with water, the beginning percent composition of Unknown ____/H_2O solution would be: (show calculations!)